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The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

98430014.5

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
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Sheet 2 of the certificate
Page 2 de l'attestation

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Process for early detection of reactor fouling in gas phase polymerisation

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Case 9007/B349 (1)

PROCESS FOR EARLY DETECTION OF REACTOR FOULING IN GAS PHASE POLYMERISATION

The present invention relates to a process for early detection of reactor fouling occurring during a gas phase polymerisation. More particularly, the present invention relates to a process for early detection of reactor fouling occurring during a gas phase polymerisation of olefin(s) using a fluidized bed reactor comprising a fluidization grid.

It is known to polymerize one or more monomers in gas phase at a pressure which is higher than atmospheric pressure in a fluidized bed reactor where polymer particles being formed are maintained in the fluidized state by virtue of a reaction gas mixture containing the monomer(s) to be polymerized and travelling in an upward stream. The polymer thus manufactured in powder form is generally drawn off from the reactor in order to maintain the bed at a more or less constant volume. The process according to the present invention employs a fluidization grid which distributes the reaction gas mixture through the bed. The reaction gas mixture leaving via the top of the fluidized bed reactor is recycled to the base of the latter under the fluidization grid through the intermediacy of an external circulation conduit fitted with a compressor.

In general, the fluidized bed reactors according to the present invention can be represented by a volume whose enclosure (wall) consists of at least one surface of revolution generated by the rotation of a rectilinear and/or curvilinear segment about a vertical axis known as an axis of revolution, and of a disengagement vessel which is above the said surface. The wall of the reactor is therefore a surface of revolution comprising a vertical axis of revolution above which is the enclosure of a disengagement vessel.

Conventional fluidized bed reactors employed for the gas phase polymerization of olefin(s) usually consist of a cylinder (1) with a vertical axis, comprising a fluidization grid at its basis, and above which is a disengagement vessel (3), in accordance with Figure 1, which shows diagrammatically a preferred apparatus for gas phase polymerization according to the present invention. The cylindrical part of the reactor is usually characterised by a height/diameter ratio (H/D) which is comprised between 1 and 15, preferably between 2 and 8, D representing the

internal diameter of the reactor. The disengagement vessel which is above the cylinder capable of containing the fluidized bed has, in principle, a cross-section which is larger than that of the cylinder. It is preferably in the shape of a bulb consisting essentially of a conical frustum of revolution with a vertical axis coinciding with the axis of the cylinder, with an apex pointing downwards with an angle preferably of between 10° and 60° and having above it a dome of substantially hemispherical shape. The small base of this conical frustum coincides with the upper end of the cylinder of the reactor, and its large base coincides with the base of the dome. It may also consist of a vertical cylinder connected to the cylinder capable of containing the fluidized bed by a connecting surface in the shape of a flared conduit. In this case this cylinder has a vertical axis coinciding with the axis of the cylinder capable of containing the fluidized bed and a roof generally of substantially hemispherical shape.

The essential purpose of the disengagement vessel is to slow down the upward gas stream which, after having passed through the fluidized bed, can entrain relatively large quantities of solid particles. As a result of this, most of the entrained solid particles return directly into the fluidized bed. Only the finest particles can be entrained out of the reactor.

Many malfunctions can occur during a gas phase polymerisation. The major consequence of those malfunctions is the generation of agglomerates in the reactor. It can affect the properties of the produced polymer. It can further affect the fluidization characteristics of the fluidization gas, which can lead to channelling and potential irreversible problems.

This agglomerations formation can occur at any time in the reactor following one or a number of different malfunctions.

As example, a malfunction may create the formation of agglomerates by adhesion of molten particles of catalyst and of polymer to the wall of the reactor, inter alia in the disengagement vessel. Agglomerations formation are usually referred to in the prior art as fouling of the reactor.

When these agglomerates become heavy they can separate from the wall and thus block the fluidization grid and/or the system for withdrawing the polymer.

To prevent the fouling of the reactor from affecting the operation of the polymerization system and the quality of the polymer produced, the reactor is

stopped at regular intervals, in order to clean the reactor walls and to extract the agglomerates. This can be done by means of water or nitrogen under pressure. Cleaning of this type brings about the introduction of poisons into the reactor, requiring extensive purges of the reactor and drying in order to remove these poisons. This procedure takes time and is not very economical.

There are a lot of disclosures in prior art of those fouling phenomena as well as many different tentative explanations for its occurrence. Sometimes the type of catalyst used is said to be responsible for the fouling ; static electricity has also been indicated as being a cause thereof ; operating conditions have also been considered as the most important criteria ; in fact, the man in the art has developed many different theories and proposals for explaining and trying to reduce fouling phenomena.

It would be a major improvement in the art if a simple method was available for early detection of reactor fouling. An early detection of those problems would then allow to take an early active step in order to mitigate or even eliminate said problems. Rapidity of action is indeed crucial in the present technology.

The Applicants have now unexpectedly found a method for detection at the early stage of reactor fouling occurring during gas phase polymerization using a fluidized bed reactor comprising a fluidization grid.

The present invention consequently consists of a process for early detection of reactor fouling occurring during a gas phase polymerisation using a fluidized bed reactor comprising a fluidization grid, characterized in that the upper part of the fluidization grid is fitted with devices capable of detecting the polymer agglomerates falling on and/or hitting said devices.

Although not wishing to be limited to one type of polymerization in particular, the present invention is particularly appropriate to the polymerization reactions of one or more of the monomers such as olefins, polar vinyl monomers, dienes, acetylenes and aldehydes.
The process according to the present invention preferably applies to the polymerization of one or more olefinic monomers such as ethylene, propylene, 1-butene, 1-pentene, 4-methyl-1-pentene, 1-hexene and 1-octene.

The devices used according to the present invention can be any device capable of detecting the polymer agglomerates which fall on it.
According to a preferred embodiment of the present invention, the devices comprise switches which protrude through the grid ; when a polymer agglomerate hits a switch, said switch gives an indicative signal of the presence of agglomerates on the grid ; there is then, preferably, an automatic reset in order to allow the switch to give a further signal in case of another encounter with an agglomerate hitting the switch.

The devices can be of a mechanical, electrical, electromechanical or pneumatic type, or made by one or more of the said types. The devices are preferably chosen so that they are not influenced by the polymerisation conditions such as temperature and pressure occurring in the reactor. The devices are preferably electromechanical.
Preferred devices are similar or identical to the Telemecanique (Schneider Group) switches sold under the trade name XCK. Such switches are typical position detectors used in the automatised installations and based on a mechanical action on electrical contacts. The Telemecanique XCK-P switches in accordance with CENELEC EN 50047 norm are ideally used.

The fluidization grid is thus fitted with devices capable of detecting the polymer agglomerates hitting said devices. The amount and size of devices used depend obviously of the size of the grid, the size of the reactor and the characteristics of the polymer agglomerates to be detected. These devices can be identical or different depending on their position on the grid.
The present invention is particularly useful for the industrial gas phase olefin(s) polymerisation using a fluidized bed reactor comprising a fluidization grid which is a disk having a diameter preferably comprised between 2 and 7 m , more preferably between 4 and 6 m. The fluidization grid is preferably fitted with at least 4 devices capable of detecting the polymer agglomerates, more preferably at least 10 devices.

Contrary to the legitimate fears which a person skilled in the art in the field of fluidized bed polymerisation might have had when using the said devices according to the present invention, the Applicant Company has quite unexpectedly found that not only did the process provides an easy and efficient method for the early

detection of polymer agglomerates falling on the fluidization grid, and thus for the early detection of malfunctions occurring during a gas phase polymerisation using a fluidized bed reactor comprising a fluidization grid,
but also that the process could be applied without any influence on the grid performance and on the fluidization characteristics.

Figure 1 shows diagrammatically an illustration of an apparatus for gas phase polymerization of olefin(s) according to the present invention. The apparatus includes:

(i) a fluidized bed reactor (1) fitted with a top (2) and a base comprising a fluidization grid (4), and consisting of a cylinder with a vertical side wall and a disengagement or desurging chamber (3) above the said cylinder, the top of the chamber (3) forming the top (2) of the reactor,

(ii) an entry chamber (9) for a reaction gas mixture, situated under the grid (4) and communicating with the reactor (1) through the intermediacy of the grid (4),

(iii) an external circulation conduit (5) for the reaction gas mixture, connecting the top (2) of the reactor to the entry chamber (9) for the reaction gas mixture and including a compressor (8) and at least one heat exchanger (6, 7), and

(iv) devices (for the early detection of agglomerates falling on the grid) protruding through the fluidization grid.

One of more conduits (10) for feeding the reaction gas mixture with constituents such as one or more olefins, for example ethylene or propylene or C4 to C10 alpha-olefins, one or more, preferably unconjugated, dienes, hydrogen and one or a number of inert gases such as nitrogen or C1 to C6 (preferably C2 to C5) alkanes such as e.g. ethane or propane may open into the external circulation conduit (5).

The process according to the invention is very particularly suitable for manufacturing polyolefins in powder form, in particular of high or linear low density polyethylene, of relative density ranging, for example, from 0.87 to 0.97, or of polypropylene, or of copolymers of propylene with ethylene and/or C4 to C8 olefins, or of elastomeric copolymers of propylene with ethylene and optionally at least one unconjugated diene, of relative density ranging, for example, from 0.85 to 0.87.. The polymers manufactured according to the present process may in

particular be powders corresponding essentially to the B type and sometimes to the A and B types, according to the classification given by D. Geldart in "Gas Fluidization Technology" published in "A. Wiley-Interscience Publication" by John-Wiley & Sons (1986), pages 33 to 46. The polymers may consist of particles which have a mass-average diameter ranging from 300 to 2000, preferably from 500 to 1500 μm.

The process for gas phase continuous polymerization of olefin(s) is carried out in a reactor containing a fluidized and optionally mechanically stirred bed maintained at an absolute pressure P1 which can range from 0.5 to 6, preferably from 1 to 4 MPa. The temperature of the fluidized bed may be maintained at a value ranging from 30 to 130°C, preferably from 50 to 110°C. A gas reaction mixture passes through the reactor at an upward speed which may range from 0.3 to 1 m/s, preferably from 0.4 to 0.8 m/s. The reaction gas mixture may contain one or more olefins, especially from C2 to C10, preferably from C2 to C8, for example ethylene or propylene, or a mixture of ethylene with at least one C3 to C10, preferably C3 to C8, olefin, for example propylene, 1-butene, 1-hexene, 4-methyl-1-pentene or 1-octene and/or else with at least one diene, for example an unconjugated diene. It may also contain hydrogen and/or an inert gas such as nitrogen or an alkane, for example from C1 to C6, preferably from C2 to C5. It may be carried out in the presence of a catalyst comprising at least one transition metal belonging to groups 4, 5 or 6 of the Periodic Classification of the elements (approved by the Nomenclature Committee of the "American Chemical Society", see "Encyclopedia of Inorganic Chemistry", editor R. Bruce King, published by John Wiley & Sons (1994)). In particular, a catalyst system of the Ziegler-Natta type may be employed, including a solid catalyst comprising a compound of a transition metal such as those mentioned above and a cocatalyst comprising an organometallic compound of a metal belonging to groups 1, 2 or 3 of the Periodic Classification of the elements, for example an organoaluminium compound. High activity catalyst systems have already been known for many years and are capable of producing large quantities of polymer in a relatively short time, with the result that it is possible to avoid the stage of removal of the catalyst residues present in the polymer. These high activity catalyst systems generally include a solid catalyst comprising essentially transition metal, magnesium and halogen atoms. It is also possible to employ a high activity catalyst essentially comprising a chromium oxide activated by a heat treatment and used in combination with a granular support

based on refractory oxide. The polymerization process is very particularly suitable for being employed with metallocene catalysts such as zirconocene, hafnocene, titanocene or chromocene, or Ziegler catalysts supported on silica, for example based on titanium or vanadium. The abovementioned catalysts or catalyst systems may be employed as they are directly in the fluidized bed reactor or may be converted beforehand into olefin prepolymer, in particular in the course of a prepolymerization bringing the catalyst or catalyst system into contact with one or more olefins such as those referred to above, in a hydrocarbon liquid medium or in gaseous phase, according, for example, to a noncontinuous or continuous process. For example, the polymerisation reaction may be carried out using a metallocene type catalyst such as those described in EP 0 129368, EP 0 206794, EP 0 420436 and EP 0 416815 the subject matter of which is incorporated herein by reference. It is also possible to use late transition metal e.g platinum or palladium, catalyst complexes such as those described in WO 9623010 the subject matter of which is incorporated herein by reference.

The polymerisation reaction may be conducted in a reactor containing a fluidised bed by techniques known per se and using equipment such as that described in French patent No. 2 207 145, French patent No. 2 335 526, EP-B-0 699213, EP-B-0 89691 or US 5352749, the disclosure of which are incorporated herein by reference.

The advantages of the process according to the invention are numerous. In fact, the process is not only simple, reliable and easy to implement, but it also makes it possible by early detection of reactor fouling occurring during the polymerization to react rapidly and thus minimise and/or radically eliminate the problems associated with reactor fouling.

The experience shows that the process according to the present invention allows operating of a gas phase reactor in a more safe and reliable way.
Another very important advantage associated with the use of the process according to the present invention is that it allows the difference between reversible and potentially irreversible fouling problems to be identified. As already explained above, there are many different methods in the art which were developed in order to take corrective actions to fouling phenomenon ; such methods are based on a hypothetical scientific explanation of the fouling phenomenon and its

corresponding measurement, e.g. temperature probe measurement or static probe measurement as disclosed in EP-224479. The fouling phenomenon measurement disclosed in the prior art relates to highly localised phenomena which are not necessarily representative of the overall phenomena occurring in the total reactor vessel. This problem associated with the prior art methods is now solved with the process according to the present invention.

Indeed, according to the present invention, a reversible fouling problem is detected when one or more switches give the indicative signal (alarm) corresponding to an agglomerate falling on the said switches and then reset automatically (i.e. went back to its original position). This may happen some time during polymerisation without leading to highly detrimental problems and without necessarily inducing any corrective action. If this appears to happen more often, i.e. with an increasing frequency, then those frequent agglomerate falls on the grid are representative of a more serious fouling problem that might be solved by applying corrective actions, e.g. by fine tuning the process operating conditions.

According to the present invention, a potential irreversible fouling problem is detected when one or more switches give the indicative signal (alarm) corresponding to agglomerates falling on the said switches and then do not reset automatically (i.e. do not return to its original position). This kind of detection is representative of a very important fouling problem which requires an urgent corrective action to be taken.

In view of the hereabove specific embodiment of the present invention, it is obvious that the present invention represents a very valuable tool for the polymerisation industry. It is also clear that the present invention is not limited to the specific embodiments hereindisclosed.

For example, depending on the number of detecting devices protruding through the grid and further to some complementary experimentation, it is even possible to determine the amounts and sizes of the agglomerates falling on the fluidisation grid, i.e. to build a kind of digital print of the fouling phenomenon occurring in the reactor.

Case 9007/B349 (1)

Claims

1. Process for early detection of reactor fouling occurring during a gas phase polymerisation of olefin(s) using a fluidized bed reactor comprising a fluidization grid, characterised in that the upper part of the fluidization grid is fitted with devices capable of detecting the polymer agglomerates hitting said devices.

2. Process according to claim 1 wherein the devices comprise detection switches which protrude through the grid and which gives an indicative signal of the presence of agglomerates on the grid.

3. Process according to any of the preceding claims wherein the devices comprise an automatic reset system in order to allow a device to give a further signal in case of another encounter with an agglomerate hitting said device.

4. Process according to any of the preceding claims wherein the devices are of a mechanical, electrical, electromechanical or pneumatic type, or made by one or more of the said types.

5. Process according to claim 4 wherein the devices are electromechanical.

6. Process according to any of the preceding claims wherein the gas phase polymerisation olefin(s) uses a cylindrical fluidized bed reactor comprising a fluidization grid which is a disk having a diameter comprised between 2 and 7 m.

7. Process according to any of the preceding claims wherein the fluidization grid is fitted with at least 4 devices capable of detecting the polymer agglomerates.

8. Apparatus for the gas phase polymerisation of olefins including :
   (i) a fluidized bed reactor (1) fitted with a top (2) and a base comprising a fluidization grid (4), and consisting of a cylinder with a vertical side wall and a disengagement or desurging chamber (3) above the said cylinder, the top of the chamber (3) forming the top (2) of the reactor,
   (ii) an entry chamber (9) for a reaction gas mixture, situated under the grid

(4) and communicating with the reactor (1) through the intermediacy of the grid (4), and

(iii) an external circulation conduit (5) for the reaction gas mixture, connecting the top (2) of the reactor to the entry chamber (9) for the reaction gas mixture and including a compressor (8) and at least one heat exchanger (6, 7),

characterised in that the upper part of the fluidization grid is fitted with detection devices protruding through the fluidization grid and capable of detecting the polymer agglomerates hitting said devices.

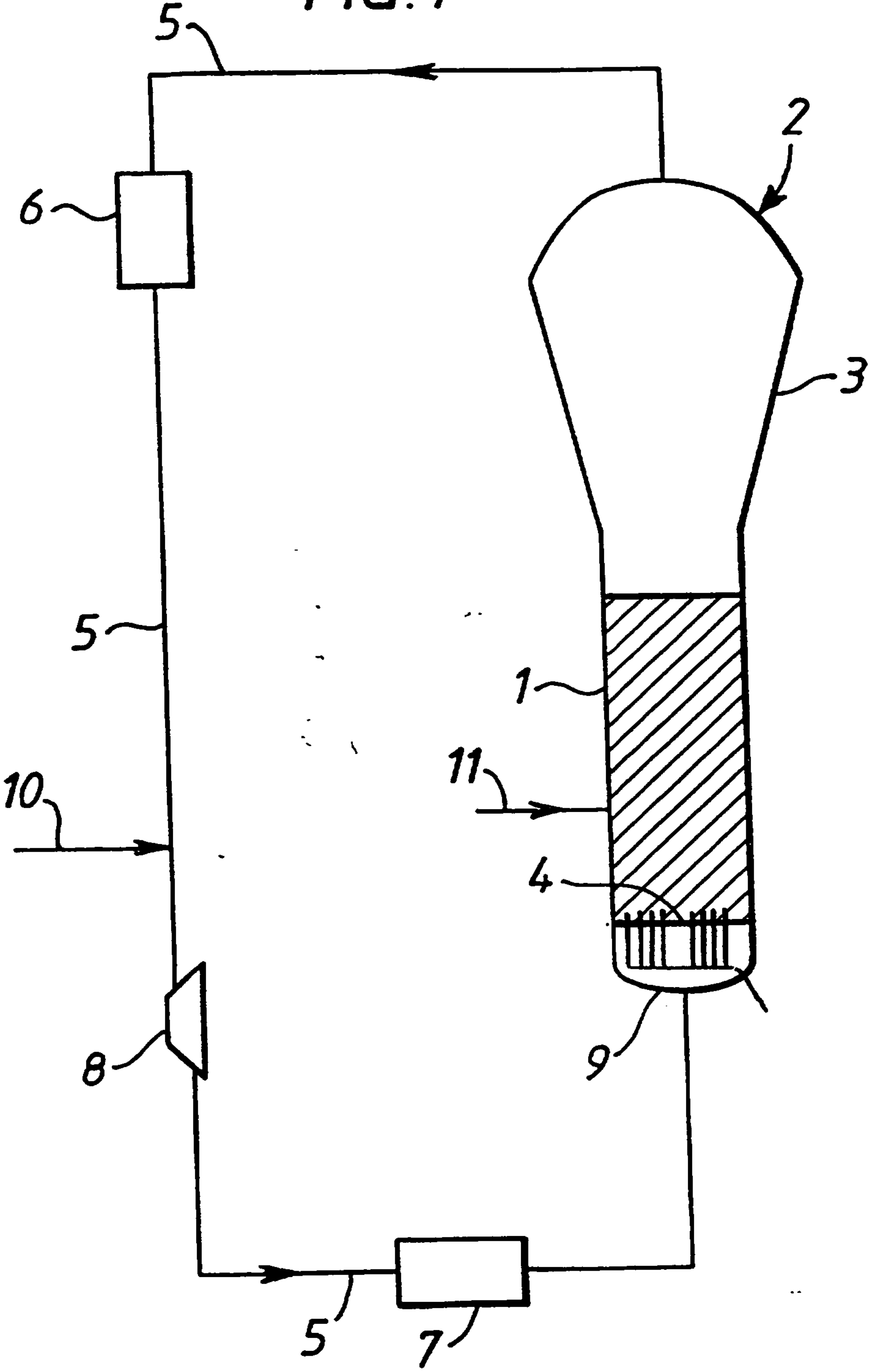
Case 9007/B349 (1)

Abstract

PROCESS FOR EARLY DETECTION OF REACTOR FOULING IN GAS PHASE POLYMERISATION

The present invention relates to a process for early detection of reactor fouling occurring during a gas phase polymerisation of olefin(s) using a fluidized bed reactor comprising a fluidization grid fitted with detection devices.

FIG. 1



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Anmelder/Applicant/Demandeur/Patentinhaber/Proprietor/Titulaire
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Kosten für Kopien/Cost of copies/Frais pour copies (____ Blätter) pages) feuilles)	0,00	0,00
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c) durch Übergabe oder Übersendung v n Schecks, die an die Order des Amts lauten,

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Under Art. 5 of the rules relating to Fees the fees may be paid as follows:

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The list of bank and giro accounts opened in the name of the European Patent Organisation and corresponding currencies for payment is reproduced on Form 2566.2.

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List of bank and giro accounts opened in the name of the European Patent Organisation and corresponding currencies for payment

Liste des comptes bancaires et de chèques postaux ouverts au nom de l'Organisation européenne des brevets et des monnaies de paiement correspondantes

	Bankkonten Bank accounts Comptes bancaires	Postscheckkonten Giro accounts Comptes de chèques postaux	Zahlungswährung Currency for payment Monnaies de paiement
AT	N° 102-133-851/00 (BLZ 12000) Bank Austria AG Am Hof 2 A-1010 Wien	N° 7451.030 Österreichische Postsparkasse Georg-Coch-Platz 2 A-1018 Wien	Österr. Schilling (ATS/EUR)
BE	N° 310-0449878-78 Banque Bruxelles Lambert BP 948 B-1000 Bruxelles	N° 000-1154426-29 Banque de la Poste B-1100 Bruxelles	Franc belge (BEF/EUR)
CH	N° 322 005 01 B UBS CH-8021 Zürich	N° 30-30796-1 Zahlungsverkehr PTT Verarbeitungszentrum CH-4040 Basel	Franc suisse (CHF)
CY	N° 0155-06-000-650 Bank of Cyprus 21, Evagoras Av, P. O. Box 1472 CY - 1599 Nicosia		Cyprus Pound (CYP)
DE	N° 3 338 800 00 (BLZ 700 800 00) Dresdner Bank Promenadeplatz 7 D-80273 München	N° 300-800 (BLZ 700 100 80) Postbank München D-80318 München	Deutsche Mark (DEM/EUR)
DK	N° 3015133759 Den Danske Bank Holmens Kanal Dept. Holmens Kanal 2 DK-1090 Kobenhavn K.	N° 899-5893 GIROBANK A/S Girostroget 1 DK-0800 Hoje Taastrup	Danske kroner (DKK)
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FI	N° 200118-182076 Merita Bank Senaatintori FIN-00020 Merita	N° 800013-90405 Leonia Fabianinkatu 23 FIN-00007 Helsinki	Suomen Markka (FIM/EUR)
FR	N° 200 20463, Code banque 30 004, Code guichet 00 567, Cle Rib 29 Banque Nationale de Paris Agence France-Etranger 2 Place de l'Opéra F-75002 Paris		Franc français (FRF/EUR)
GB	N° 60 271 489 (sorting-code 20-00-00) Barclays Bank PLC 54 Lombard Street P.O. Box 544 GB-London EC3V 9EX		Pound Sterling (GBP)
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Patentanmeldung Nr. Patent application No. Demande de brevet n°

98430013.7

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
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Case 9004/B348 (1)

Polymerisation process

The present invention relates to a process for the gas phase polymerisation of olefins in a fluidised bed reactor and in particular to the introduction of a deactivating agent into the reactor without terminating the polymerisation reaction.

It is known to polymerise one or more alpha-olefins, such as ethylene or propylene, in the gas-phase in a fluidised bed reactor, in the presence of a catalyst based on a transition metal belonging to the groups IV, V or VI of the Periodic Table of the Elements; in particular in the presence of a catalyst of the Ziegler-Natta type, chromium oxide type or a metallocene catalyst. Catalyst particles, growing and formed polymer particles are kept in the fluidised and/or agitated state in a gaseous reaction mixture containing the alpha-olefin or alpha-olefins, which are introduced continuously into the reactor. The catalyst is introduced continuously or intermittently into the reactor while the polymer constituting the fluidised bed is withdrawn from the reactor, also continuously or intermittently. The heat of the polymerisation reaction is essentially removed by the gaseous reaction mixture, which may be passed through a heat transfer means before being recycled into the reactor.

Fouling of the reactor wall is a well-known phenomenon in gas phase polymerisation processes. Hot spots and hence fouling are likely to occur in the vicinity of, especially at or close to the reactor walls. Where a polymerisation reactor is equipped with a fluidisation grid the hot spots/fouling are most typically likely to occur in the region 0.25D to 0.75D above the grid (where D is the diameter of the reactor). Hot spots/fouling may also occur in the disengagement zone. Such hot spots/fouling are usually detected by the use of thermocouples or temperature indicators either attached to the reactor wall or inserted a small distance into the reactor itself. Such detection means may be associated with means for recording temperature variations and possibly visual and/or audible alarms.

The explanations for the occurrence of fouling are many and varied. Operating conditions, even small variations in the reaction conditions have been considered important to the occurrence of fouling. For example, poor or loss of fluidising gas flow can cause catalyst particles, formed polymer particles and growing polymer particles to be insufficiently cooled by the gaseous reaction mixture passing through the reactor. This insufficient heat removal gives rise to hot spots, which can lead to the melting of the catalyst/polymer particles resulting in the formation of agglomerates of molten catalyst/polymer which then may melt on and cause fouling of the reactor walls. Other reasons given for the occurrence of fouling include the type of catalyst used and the presence of static electricity. Whatever the explanation of its occurrence the presence of fouling very often induces fluidisation pertubations which can lead to irreversible process problems. For example, when the agglomerates become heavy, they can come off the wall and block the fluidisation grid and/or the polymer withdrawal system. Severe hot spot and therefore fouling problems may necessitate a shut-down of the reactor. One way in which the polymerisation process can be terminated quickly to avoid irreversible process problems and/or a reactor shut down is to introduce a deactivating agent whose purpose is to kill or terminate the fluidised bed polymerisation.

European Patent EP-B-0 471 479 discloses a process for completely stopping a gas-phase alpha-olefin polymerisation reaction carried out with the aid of a chromium oxide-based catalyst by introducing a deactivating agent such as oxygen, ammonia or carbon monoxide into the polymerisation reactor

US Patent No. 4,306,044 discloses a system for introducing carbon dioxide into a gas-phase olefin polymerisation process to at least reduce the rate of the polymerisation reaction.

Thus there remains the need to reduce or even eliminate hot spots/fouling in gas-phase olefin polymerisation processes without terminating the polymerisation reaction.

It has now been unexpectedly found that hot spots and hence fouling can be reduced or even eliminated by using the process according to the present invention without the need to stop production or shut down the reactor.

Thus, according to the present invention there is provided a process for the gas phase polymerisation of one or more alpha-olefins in the presence of a polymerisation catalyst system under reactive conditions in a fluidised bed reactor which process comprises introducing into the reactor a deactivating agent characterised in that the deactivating agent is introduced close to the reactor wall and that said introduction does not terminate the polymerisation reaction

The deactivating agent may be selected from a wide variety of products which are known to or are capable of reducing the polymerisation rate of an alpha-olefin in the presence of a catalyst based on a transition metal e.g a catalyst of the Ziegler-Natta type or a metallocene catalyst, or a chromium oxide type catalyst. The deactivating agent can be selected especially from polymerisation inhibitors or from poisons known for this type of reaction such as carbon monoxide, carbon dioxide, carbon disulphide, carbon oxysulphide, nitrogen oxides and peroxides, oxygen, alcohols, aldehydes, ketones, thiols and water. Deactivating agents which can be selected in particular are carbon monoxide, oxygen and water, especially carbon monoxide.

The deactivating agent may be employed alone or, preferably, diluted in an inert gas such as nitrogen. When carbon monoxide is employed as deactivating agent, it may be employed in the form of a gaseous mixture with an inert gas such as nitrogen. A mixture of two or more deactivating agents may be employed.

The deactivating agent is introduced into the polymerisation reactor in a quantity which is effective to deactivate hot spots and fouling at or in close proximity to the reactor walls. The quantity of deactivating agent required will therefore usually be less than the minimum quantity theoretically necessary for terminating the polymerisation reaction. Preferably, the deactivating agent is introduced into the polymerisation reactor in an amount such that the weight ratio of the deactivating agent to the catalyst is in the range 0.00015 - 0.0011 : 1, preferably 0.00035 - 0.001 : 1, especially 0.0006 - 0.0009 : 1. For example it has been found to be possible to deactivate hot spots and fouling at or in close proximity to the reactor walls by introducing into the polymerisation reactor 0.0002 - 0.0011g of carbon monoxide per gram of catalyst. The use of a quantity

of deactivating agent which is too large would have the effect of stopping the polymerisation reaction. The minimum quantity of deactivating agent necessary for stopping a polymerisation reaction can be obtained by previous experimentation performed in a gas phase reactor working with known quantities of catalyst and of deactivating agent.

The deactivating agent may be introduced on a continuous basis but it is preferably introduced into the polymerisation reactor over a relatively short period of time typically less than 5 minutes. The period of introduction of the deactivating agent is advantageously as short as possible and is preferably shorter than one minute and more preferably shorter than 30 seconds. The deactivating agent may be introduced intermittently throughout the polymerisation reaction i.e it may be introduced as and when required to deactivate any hot spots/fouling which form throughout the polymerisation reaction. Furthermore, the feeding of catalyst and/or olefin into the polymerisation reactor need not be discontinued.

The deactivating agent is introduced at a point situated as close as possible to the reactor wall such that the deactivating agent deactivates or kills those hot spots/fouling which are at or close to the reactor wall without deactivating the entire bed. For example the deactivating agent may be introduced at reactor wall up to a distance of from 0.1D to 0.2D from the reactor wall where D is the reactor diameter. Furthermore, it may be advantageously introduced at a plurality of points, especially at two or more points close to the reactor wall. The deactivating agent may be introduced into the reactor at one or more points above or below the bed. Preferably it is introduced at a plurality of points above the bed e.g. in the disengagement zone, and/or below the bed e.g underneath a fluidisation grid. More especially it is introduced at a plurality of points underneath a fluidisation grid and up to a distance of 0.2D from the reactor wall.

The deactivating agent may be introduced into the reactor by any suitable means which provides a substantially uniform concentration of deactivating agent around at least part of the reactor wall.

In a preferred embodiment of the present invention the deactivating agent is introduced close to the reactor wall by means of a distribution ring. Distribution

rings are well-known in the art. The spacing of the distribution holes around the ring are such as to provide a uniform distribution of a gaseous material around the whole of the distribution ring.

The distribution holes in the ring can be directed in any direction e.g upwards, downwards, horizontal from the ring provided that the size of the jets is sufficiently small to avoid excessive quantities of deactivating agent being directed towards the centre of the reactor. Classical correlations and standard chemical engineering can be used to avoid this in the design procedure. Preferably the direction of the distribution ring holes is horizontal and towards the reactor wall.

Where a fluidisation grid is employed, the distribution ring may be located either above or below the fluidisation grid, preferably below the grid, especially below the grid up to a distance of 25cm below the grid and in particular 5 to 10 cm below the grid.

For a given reactor diameter, the outer diameter of the distribution ring is preferably the maximum diameter achievable notwithstanding design considerations and mechanical constraints of fitting the distribution ring immediately adjacent to the reactor wall. In particular, the outer diameter of the distribution ring may be between 0.5 D to 1.0 D, e.g 0.75 D to 1.0 D, such as 0.90 D to 1.0 D, where D is the diameter of the reactor.

Irrespective of the whether the distribution ring is above or below a fluidisation grid the distribution ring preferably provides an essentially uniform concentration of deactivating agent in the immediate vicinity of the reactor wall.

In a particularly preferred embodiment of the present invention the deactivating agent is introduced into the reactor by means of a distribution ring having distribution holes which are horizontal and directed towards the reactor wall. The distribution ring has an outer diameter of between 0.90 D and 1.0 D (where D is the reactor diameter) and when a fluidisation grid is used, the distribution ring is positioned 5 to 10 cm below the grid.

The polymerisation reaction may be conducted in a reactor containing a fluidised bed by techniques known per se and using equipment such as that described in French patent No. 2 207 145, French patent No. 2 335 526, EP-B-0

699213, EP-B-0 89691 or US 5352749, the disclosure of which are incorporated herein by reference. The apparatus can comprise essentially a fluidised-bed polymerisation reactor comprising a vertical cylinder equipped with a fluidisation grid and supporting a disengagement chamber, a conduit for recycling the gas mixture, connecting the top of the disengagement chamber to the base of the reactor, which recycle conduit is provided with at least one heat exchanger and a gas compressor and with a conduit for introducing alpha-olefins. During the course of the polymerisation the bed comprises catalyst particles, growing polymer particles and formed polymer particles. The bed is maintained in a fluidised state by introducing a fluidising medium at a sufficient flow rate to cause the particles to separate and act as a fluid. The fluidising medium may consist of a single phase e.g a gas phase or it may be two phase for example it may consist of a gas phase and a liquid phase, for example, a mixture of gas and entrained liquid. In such a case the quantity of liquid in the gas phase may be about from 1 - 50 weight percent, for example 10-20 weight percent, preferably less than 10 weight percent, provided always that the velocity of the two-phase fluidising medium is high enough to keep the liquid phase in suspension in the gas and to support the fluidised bed in the reactor. The two-phase fluidising medium of gas and entrained liquid may be formed by cooling part or all of the unreacted fluids from the reactor below the dew point and re-introducing said two-phase mixture into the reactor. Alternatively a two-phase fluidising medium may be formed within the reactor, for example by separately injecting e.g underneath a fluisisation grid or directly into the fluidised bed, gas and liquid under conditions which will produce a two-phase fluidising medium. A single phase fluidising medium or a substantially single phase fluidising medium such as a gas phase fluidising medium may be formed by cooling a recycle gaseous stream to a temperature sufficient to form a liquid and a gas and by separating the liquid from the gas and then feeding the gas into the reactor, for example by injection below the fluidisation grid.

Alternatively, the recycle gaseous stream may be divided into a first stream and a second stream. The first stream is passed directly to the reactor in a conventional way by injection below the fluidisation grid and the second stream is

cooled and the stream is separated into a gas and liquid stream. The gas stream may be returned to the first stream and re-introduced into the reactor below the bed, for example, below the fluidisation grid if such a grid is employed.

The fluidising medium may contain unreacted gaseous monomers, make-up monomers to replace monomers consumed by the polymerisation reaction, and optionally inert hydrocarbons e.g ethane, propane, butane, isobutane or isopentane, inert gases such as nitrogen, reaction activators or moderators e.g hydrogen.

The fluidisation grid positioned above the point of recycle ensures proper distribution of the fluidising medium to the bed and acts as a support for the bed when the supply of gas is cut off.

The polymerisation reaction may be carried out in the presence of catalysts of the Ziegler-Natta type based on magnesium, halogen, titanium and/or vanadium and/or zirconium. The Ziegler-Natta type catalyst can be used directly as such or in the form of a prepolymer. The conversion to prepolymer is generally carried out by bringing the catalyst into contact with one or more alpha-olefins in amounts such that the prepolymer contains between 0.002 and 10 millimol of transition metal per gram

A co-catalyst may also be used with the Ziegler-Natta catalyst such as organometallic compounds of metals belonging to Groups I, II or III of the Periodic Table of the elements, in particular organoaluminium compounds.

The polymerisation reaction may also be carried out using a metallocene type catalyst such as those described in EP 0 129368, EP 0 206794, EP 0 420436 and EP 0 416815 the subject matter of which is incorporated herein by reference.

It is also possible to use late transition metal e.g platinum or palladium, catalyst complexes such as those described in WO 9623010 the subject matter of which is incorporated herein by reference.

The polymerisation reaction is generally carried out under a pressure of 0.5 to 6 MPa and at a temperature of 30°C to 135 °C. The process is suitable for the polymerisation of one or more alpha-olefins containing 2 to 8 carbon atoms, in particular for the polymerisation of ethylene or propylene. It is especially suitable for the copolymerisation of ethylene with at least one alpha-olefin containing from

3 to 8 carbon atoms, or for the copolymerisation of propylene with at least one alpha-olefin containing from 4 to 8 carbon atoms.

The process according to the present invention will now be illustrated with reference to the accompanying drawings.

Figure 1 is a cross-sectional view of a cylindrical polymerisation reactor

Figure 2 is a front view of the lower part of a cylindrical polymerisation reactor

Figure 1 illustrates part of a cylindrical polymerisation reactor comprising a reactor (1) of diameter (D), an entry point (3) into the reactor (1) for the deactivating agent and a distribution ring (2) of diameter fD

Figure 2 illustrates the lower part of a polymerisation reactor comprising a reactor wall (1), a fluidisation grid (6) and below the fluidisation grid (6) a distribution ring (2). The fluidising gas enters the reactor (1) through entry point (7) and moves upwards towards and through the fluidisation grid (6). Deactivating agent which enters the reactor (1) at entry point (3) is transported through the distribution ring (2) by the upwardly flowing fluidising gas and into the deactivating zones (4). No or little deactivating agent is transported into zone (5)

The following Examples will illustrate the present invention

<u>Example 1</u>

The process is carried out in a fluidised bed gas phase polymerisation reactor consisting of a vertical cylinder of diameter 0.75m and height 5 m and surmounted by a velocity reduction chamber. In its lower part, the reactor is equipped with a fluidisation grid and an external line for recycling gas, connecting the top of the velocity reduction chamber to the lower part of the reactor, situated under the fluidisation grid. The gas recycling line is equipped with a compressor

and with a heat transfer means. Opening into the gas recycling line there are, in particular, the feed lines for ethylene, 1-butene, hydrogen and nitrogen, which represent the main constituents of the gas reaction mixture passing through the fluidised bed.

The distribution ring has an outer diameter of 0.705 m and is located 6 cm below the fluidisation grid. A schematic description of said distribution ring is given in Figures 1 and 2.

Above the fluidisation grid the reactor contains a fluidised bed consisting of a linear low density polyethylene powder. The gas reaction mixture, which contains ethylene, 1-butene, hydrogen and nitrogen passes through the fluidised bed at a pressure of 2 MPa, at 80 °C and with an upward fluidisation velocity of 0.36 m/s.

The polymerisation reactor is fitted with three sets of wall temperature indicators at 0.5m 1.0m and 1.5m above the fluidisation grid.

The catalyst used is a Ziegler-Natta catalyst prepared according to Example 1 of EP-A- 0 529 977.

Under these conditions a linear low density polyethylene with a density of 0.918, a melt index of 0.9g/10 minutes under a 2.16-kg load at 190 °C is manufactured at an output of 97 kg/h.

Following a period of stable operation, for unknown reasons the wall temperature indicator at the 0.5m position reached 85 °C ; in response, carbon monoxide gas diluted to a level of 10000 ppm in a nitrogen gas carrier was introduced into the reactor via the distribution ring. The quantity of carbon monoxide injected was 0.00025 g of CO per g of catalyst present in the bed. Within a period of approximately 30 seconds all of the wall temperature indicators were seen to stop increasing and within 5 minutes had returned to their nominal values of 80 °C. During this period the feeding of catalyst into the reactor was not discontinued and no production rate change was observed.

Case 9004/B348 (1)

Claims

1. A process for the gas phase polymerisation of one or more alpha-olefins in the presence of a polymerisation catalyst system under reactive conditions in a fluidised bed reactor which process comprises introducing into the reactor a deactivating agent characterised in that the deactivating agent is introduced close to the reactor wall and that said introduction does not terminate the polymerisation reaction.

2. A process according to claim 1 wherein the deactivating agent is introduced at the reactor wall up to a distance of 0.2D from the reactor wall where D is the diameter of the reactor.

3. A process according to any one of claim 1 or claim 2 wherein the deactivating agent is introduced at one or more points below a fluidisation grid.

4. A process according to any one of claim 1 or claim 2 wherein the deactivating agent is introduced at one or more points above the bed.

5. A process according to any one of the preceding claims wherein the deactivating agent is introduced in a substantially uniform concentration around at least part of the reactor wall.

6. A process according to any one of the preceding claims wherein a distribution ring is employed to introduce the deactivating agent.

7. A process according to claim 6 wherein the distribution ring has an outer diameter of from 0.50D to 1.0D where D is the diameter of the reactor.

8. A process according to claim 6 or claim 7 wherein the distribution ring is located below the fluidisation grid up to a distance of 25 cm below the grid.

9. A process according to any one of the preceding claims in which the deactivating agent is introduced in a weight ratio of deactivating agent to catalyst of 0.00015-0.0011 : 1.

10. A process according to any one of the preceding claims in which the deactivating agent is a polymerisation inhibitor.

11. A process according to claim 10 wherein the polymerisation inhibitor is selected from carbon monoxide, water and oxygen.

12. A process according to any one of claim 1 to claim 8 wherein the deactivating agent is carbon monoxide and is introduced in a weight ratio of carbon monoxide to catalyst of 0.0002-0.0011 : 1.

13. A process according to any one of the preceding claims wherein the catalyst is a catalyst of the Ziegler-Natta type based on magnesium, halogen, titanium and/or vanadium and/or zirconium.

14. A process according to any one of claims 1 to 12 wherein the catalyst is a metallocene catalyst.

15. A process according to any one of the preceding claims wherein the alpha-olefin(s) contain from 2 to 8 carbon atoms.

16. A process according to any one of the preceding claims wherein the gas phase polymerisation is carried out according to EP-B-0 699213 or EP-B-0 89691 or US 5352749.

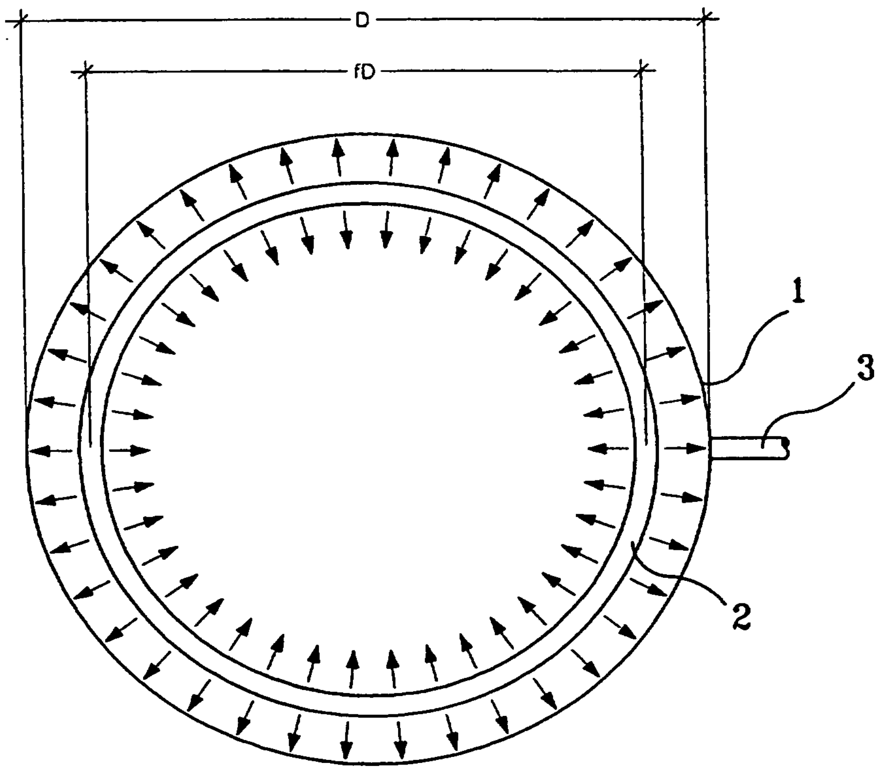
Case 9004/B348 (1)

Abstract

Polymerisation process

A process for the gas phase polymerisation of one or more alpha-olefins in the presence of a polymerisation catalyst system under reactive conditions in a fluidised bed reactor which process comprises introducing into the reactor a deactivating agent characterised in that the deactivating agent is introduced close to the reactor wall and that said introduction does not terminate the polymerisation reaction.

FIG.1



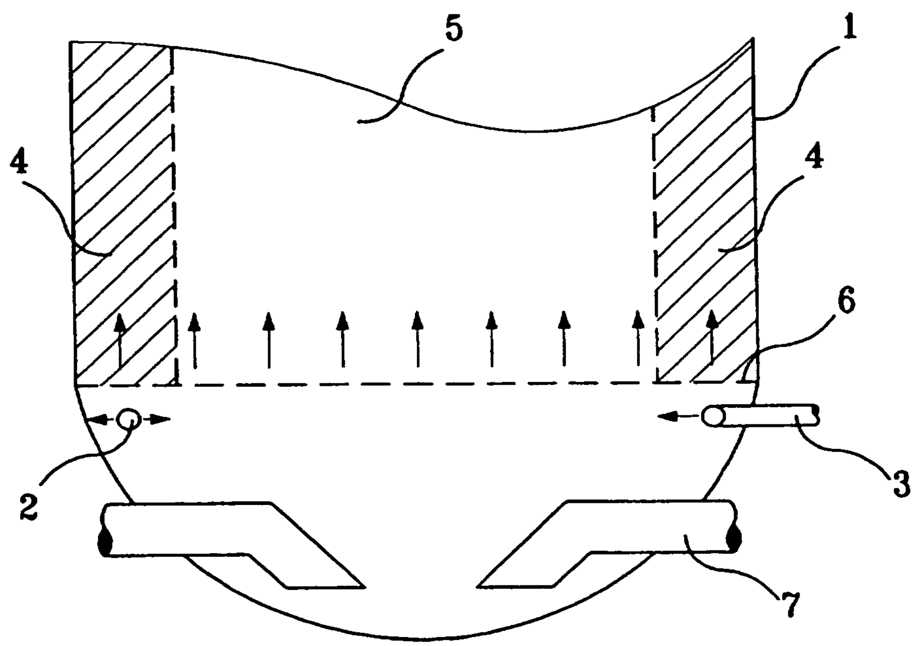


FIG.2